THE SOLVENT EXTRACTION OF WAX
FROM SORGHUM BRAN

by

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TABLE OF CONTENTS

INTRODUCTION

The ultimate aim of this project[1], which has been under investigation since 1946 at Kansas State College, is the selection and successful operation of a low capacity extraction unit suitable for extracting the wax from sorghum bran. The problem is a complex one for several reasons such as:

1. The wax fraction (esters of long chain monocarboxylic acids and long chain alcohols) of the bran comprises only about two percent of its weight. Such a small weight fraction of desirable product presents problems in the handling of large quantities of inert material. Also, in order that an extraction process be economical, it is evident that virtually all of the wax will have to be extracted allowing only traces to remain in the solids leaving the extractor.

2. Sorghum oil (glyceral esters of various fatty acids) is obtained to an appreciable extent as a by-product making necessary an expensive separation of the wax from the oil. In general, the oil content of the bran will vary from one to two times the wax content; therefore, in an actual extraction, more oil is obtained than wax.

3. Sorghum wax and carnauba wax are very similar in nature and it is without a doubt that sorghum wax would

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demand very nearly the same price as the current price for
carnauba wax. The price of carnauba wax fluctuates greatly.
With such a price variation, it is possible that an ex-
traction plant could intermittently make and lose money
depending upon the demand for sorghum wax.

Sorghums have long been an old standby as far as crops
are concerned in the area of the Middle-West because of
their ability to withstand the hot, dry climate for which
most of the region is noted. Unlike corn and wheat, sorghum
requires the minimum amount of moisture and can stand up
against disease and insects in a much more resistant manner.
Indeed, with the wheat mite destroying hundreds of acres of
wheat throughout Kansas, Nebraska, and Colorado this year,
many farmers are thinking of switching their main crop to
sorghum because of its ruggedness (12).

It is a known fact that the hotter the growing season
the greater will be the wax content of sorghum bran. The
bran forms the hull or outer coating for the interior of the
whole grain comprising the endosperm and germ. In order to
protect itself from the hot penetrating rays of the sun, the
sorghum plant excretes wax to the hull. The amount of wax
excreted depends upon the heat of the summer; thus a hot,
dry summer will yield sorghum bran having a greater wax con-
tent than the bran yield of a cool, wet summer.

In carrying out the aim of the project, the author has
deemed it wise to continue where Hub (8) had left off in the

small laboratory. Additional work was done with trichloro-
ethylene as a solvent until it was realized that, despite
the fact that virtually all of the previous investigations
involved trichloroethylene, the use of that solvent was not
indispensable. Its extensive use seemed to stem almost ex-
clusively from the fact that it is non-combustible. Large
scale commercial extraction of soybean oil and cotton-seed
oil is carried out using a commercial form of hexane as the
solvent (3, 4). Although commercial hexane (henceforth re-
ferred to as Skellysolve B, a trade name) presents a very
serious safety problem because of its high flammability, ex-
traction units enclosed in vapor-tight housings, coupled
with strict safety regulations, have allowed the safe use of
the solvent on a large scale. With this in mind, the author
has done considerable work using Skellysolve B as the solvent.
The respective merits of Skellysolve B and trichloroethylene
are compared relative to this project in the Appendix of this
thesis.

The complete design of any solid-liquid extraction unit
involves knowledge of the variation of solvent hold-up (re-
tention ratio) in the solid inert material leaving each stage
of a countercurrent process. This factor has been completely
ignored heretofore. A series of experiments were run to de-
termine the retention ratio of both trichloroethylene and
Skellysolve B in the inert solids used. Using this ratio

along with other data obtained in the extraction runs, has enabled the author to predict the number of theoretical stages that will be required to effect a given extraction.

REVIEW OF PREVIOUS INVESTIGATIONS

Since the investigation into the possibility of economically extracting the wax contained in sorghum brans was begun at Kansas State College, two types of extraction apparatus have been built and tested. In 1947, Foveaux (5) built an inclined tube type of extractor modeled after the Hildebrandt unit (4). The extractor consisted of two tubes inclined with respect to one another so as to form a V. Whole grain was admitted to the top of one of the tubes and was forced to travel downward through a solvent (trichloroethylene) by means of a screw conveyor. Another screw conveyor running through the other tube branch forced the grain up this tube, again through the solvent, to the outlet where the extracted grain was expelled. Fresh solvent was continually introduced near the grain outlet and the miscella (wax-oil-solvent solution) was drawn off near the grain entrance. Hence, a continuous counter-current piece of apparatus was built and subjected to testing.

Foveaux reported a yield of extractables amounting to 0.28 percent of the weight of the grain using a temperature range of 100-142° F., a contact time of 2.70 hours, and a

solvent to grain weight ratio of 1.90. Since the bran comprises roughly about seven percent of the total grain, the yield corresponds to a yield of some four percent based on the bran fraction only, which corresponds to the maximum total extractables available in the bran used. The bran fines fraction, however, which has not been included thus far, when considered, would bring the yield down to about 2.95 percent which is more in keeping with the data obtained by the author and by previous workers in the field.

The V-shaped inclined tube apparatus was given up when it was decided that the most economical manner of extracting the wax would be to run the extraction on the bran and bran fines fractions only instead of on the whole grain as was done. This decision came about through other workers who found that the bulk of the sorghum wax was contained in the bran and bran fines fractions and the bulk of the undesirable oil by-product was contained in the germ fraction. The oil is termed here as an undesirable by-product only because the purpose of the project at hand was to develop and design a suitable extraction unit for the solvent extraction of wax from sorghum bran. Actually, the oil is valuable in its own right and would, therefore, be extracted independently of the wax extraction operation.

The decrease in the bulk density and the particle thickness of bran and bran fines as compared to that of the whole grain would conceivably cause undue plugging and clogging of

the tolerance existing between the screw blades and the extractor walls with fine suspended solid material. For this reason the inclined tube extractor was discarded as a possible extraction unit.

French (6) carried the project to the small laboratory stage and obtained much valuable rate data concerning the extraction of wax from the bran using trichloroethylene as the solvent. He also developed rate equations from which the amount of extraction could be predicted.

A Bollmann or basket type of extraction apparatus was devised and built by Medlin (9) in 1949. This piece of equipment consisted of eight baskets constructed of fifty mesh stainless steel screen which revolved clockwise about a common center by means of a variable chain drive. Each basket was filled with bran at the beginning of its descent. A spray of miscella thoroughly wetted the bran in addition to partially extracting the wax and oil. At the nadir point of the revolution, the baskets were dipped into the main bulk of miscella which was contained in the main housing of the unit. Just before the zenith point of the revolution was touched, the now-extracted bran was subjected to a spraying of fresh solvent which, of course, served to bring the percentage extraction to a maximum. Each basket was then automatically dumped and refilled for its next journey around.

Medlin found that the bulk of the extractables he obtained was wax and not oil as one would assume knowing that

the oil content of the bran he used was larger than the wax
content. He concluded from this fact that it should be
possible to effect a satisfactory separation of the wax
from the oil in the extraction step itself rather than
carry out the separation later by expensive methods.

Hub (8) carried on with Medlin's extraction unit in
the fall of 1949 making many minor changes in the construc-
tion of the unit. After several futile attempts to repro-
duce Medlin's results, Hub came to the conclusion that the
Bollmann type of extraction unit was not the one for the
particular job at hand. He pointed out that because of the
fact that the unit built was not enclosed in a vapor-tight
housing, it was virtually an impossibility to maintain the
extraction temperature high enough to do any good. With
that he brought the entire project back up to the small
scale laboratory and conducted a series of extractions in
an attempt to obtain valuable data which could be used in
the design of some other more desirable type of extraction
unit. Hub presented curves showing the effect of temperature,
time of contact, solvent ratio, and moisture content of the
bran on the amounts of wax and oil extracted. All of his
data apply when using trichloroethylene as the solvent.

MATERIALS

The trichloroethylene used in the extractions was
classified as technical grade. As taken from the drum
the solvent bore a yellow color due to the formation of
$FeCl_3$ resulting from reaction between the solvent and the
drum walls. Distillation of the solvent before use in ex-
traction produced a water clear starting material.

Skellysolve B used in the extractions was obtained
from the Chemistry Department. This solvent had an average
boiling point of 152° F. (67° C.) and was a product of the
Commercial Solvents Corporation.

The bran used is described by Medlin as follows:

> (it) was processed from country run milo graded
> as No 2 yellow. The bulk of this milo was of the
> Westland variety. It included bran from both the
> debranning and cracking operations. The debranner
> bran was cleaned by aspiration and screening over
> 30 mesh screen. The fines from the cracking bran
> were not removed.

Because this bran was nearly two years old at the be-
ginning of the author's work, it was deemed wise to secure
fresh material. New bran was ordered, but was never de-
livered; consequently, all of the data presented in this
paper are based upon this 'old' bran whose wax content (only
one percent) left much to be desired.

It became evident after considerable work with the bran
that it had been poorly milled. A substantial share of the

weight of the bran could be attributed to other fractions
of the sorghum grain. In an attempt to obtain a purer
material with which to work, the bran as received (as it
will be called throughout this paper) was separated into
two fractions using the Oliver gravity separator. These
two fractions will be termed fraction I and fraction II.
Fraction I consisted primarily of pure bran; fraction II
of germ, endosperm, debris, etc. The bran fines fraction,
usually present after the initial milling of sorghum grains,
seemed to be lacking not only in the separated fractions I
and II, but also in the bran as received. After separation
the pure bran fraction, fraction I, represented only forty
percent by weight of the original bran as received and had
a bulk density of 13.05 pounds per cubic foot.

Average moisture content of solid materials used were:
bran as received, 11 percent; fraction I, 8.54 percent;
fraction II, 8.35 percent. According to the moisture con-
tent figures given for the two separated fractions, the
bran as received should have a moisture content of about
8.40 percent. The decrease in moisture content of the sepa-
rated fractions over that of the bran received is attri-
buted to the drying effect of the air stream used during
the separation of the bran in the Oliver gravity separator.

SOXHLET EXTRACTIONS

The Soxhlet extraction apparatus is pictured on the left-hand side of Plate III. It functions by means of repeated extractions with solvent redistilled from the boiler after each pass. Each sample of material was extracted for a period of about eight hours so as to insure virtually complete extraction. The results of these extractions were taken as the wax and oil content of the material.

Average values for the wax and oil content of the bran as received and its two fractions are given in the following table as determined from a series of Soxhlet determinations:

Table 1. Extractable composition of bran fractions.

Fraction	Wax	Oil	Wax & Oil	Weight
Bran as received	1.22	2.38	3.60	100
Fraction I	1.79	1.34	3.13	40
Fraction II	1.08	4.72	5.80	60

DETERMINATION OF RETENTION RATIO OR SOLVENT HOLD-UP

As previously stated in this paper, the retention ratio or solvent hold-up of the material being extracted was heretofore ignored. These data are vitally needed if it is desired to carry out the design of extraction apparatus.

The equipment used for determining the solvent ratio
is pictured in Plate I. It consists essentially of a con-
stant-temperature water bath which surrounds a tube contain-
ing the material to be tested. The solvent, heated to the
same temperature as the solid material, is introduced at
the top of the tube and the miscella is collected in a
graduated cylinder provided beneath the tube. The solvent
reservoir shown at the right of the picture was used during
early determinations but its use had to be stopped due to
experimental inaccuracies. The main trouble presented by
the use of this reservoir was the very inaccurate measure-
ment of the volume of solvent introduced to the tube con-
taining the material to be tested. Resort had to be made to
a graduated cylinder as the solvent reservoir so that an
accurate solvent volume could be recorded.

In the calculation (explanation presented in the Appen-
dix) of the solvent ratio from the data recorded, no distinc-
tion was made between solvent and miscella. That is, the
weight of a given volume of solvent was considered equal to
the weight of the same volume of miscella. Although such an
assumption would lead to gross errors in the general extrac-
tion problem, it is a valid assumption in the case at hand.
The total percentage extractables (wax and oil) in the bran
as received averages around 3.60 as given in Table 1; conse-
quently, the weight of a given volume of miscella would not
be expected to be very different from the weight of an equal

volume of solvent provided that the solvent ratio (weight of solvent perunit weight of dry material) is not too small. The solvent ratio used in the determination of the retention ratio was high enough so that the assumption is a valid one.

The validity of the assumption is further brought out by the relationship of the line EG" to the hypotenuse of the right triangle (the line representing miscella solutions) in Figure 17. The line EG" in the figure would meet the corner of the triangle not shown if it and the two sides of the triangle were extended. This line, then, represents a constant solvent to solid ratio. If the line EG" were drawn parallel to the hypotenuse of the triangle, it would represent a constant miscella to solid ratio. Reference to the figure will immediately show that for all practical purposes no distinction could be made between the slope of the line EG" as shown and a line drawn from the origin of EG" and parallel to the hypotenuse. Thus, the terms solvent to solid ratio and miscella to solid ratio are synonymous in the type of extraction at hand.

An attempt was made to find out if the retention ratio was affected by contact time or temperature of extraction. The results indicated that within the confines of experimental error, these two variables have no effect upon the retention ratio. The effect of a change in concentration of the miscella upon the retention ratio could not be experimentally

determined because of the small amount of extractables
available from the material to be tested.

The solvent hold-up or retention ratio was found to
be substantially constant with changes in all variables
involved. The average results of many determinations are
presented in Table 2.

Table 2. Retention ratio of bran fractions.

Fraction	Solvent	Retention ratio, g solvent/g dry material
Bran as received	Skellysolve B	0.663
	Trichloroethylene	1.07
Fraction I	Skellysolve B	0.747
Fraction II	Skellysolve B	0.286

Figure 1 presents, in graphical form, the solvent hold-
up values for the bran as received using the two solvents.

EXTRACTION METHOD AND RESULTS

Plate II pictures the extraction equipment used in
making nearly one hundred runs.

So that a comparable basis could be attained, the weight
of material samples was kept within the range of 27-28 grams
on a dry basis. The volume of solvent was kept constant at
450 ml thus fixing the solvent ratio. Hence, the only vari-
ables remaining were the temperature and the contact time.
By fixing the temperature, the effect of contact time could

be studied; and by running a series of such determinations
at various temperatures, the effect of temperature on ex-
traction could be studied.

Hub (8) has presented several curves showing that, in
general, as the temperature is increased, the percentage
extraction is increased, and that as the contact time is
increased, the percentage extraction is increased. His data
were taken without the effect of agitation and are only appli-
cable to the use of trichloroethylene as the solvent. He ob-
served no selectivity of the solvent for either the wax or
the oil.

The extraction procedure was briefly as follows: Approxi-
mately 31 grams of sample were weighed out on the analytical
balance. Four hundred and fifty milliliters of solvent were
measured out and poured into the extraction vessel. The sol-
vent was then heated by means of a knife heater and variac to
a predetermined temperature. When the temperature was reached,
proper adjustment of the variac kept it constant. Agitation
was started and the material sample to be extracted was intro-
duced into the solvent. At the same time a stop-clock was
started to keep a record of the extraction time. Approxi-
mately one minute previous to the attainment of some desired
time of extraction, the contents of the extraction vessel
were vacuum filtered to separate the miscella from the solid
material. The miscella was then analyzed according to the
procedure given in a later section.

Trichloroethylene was used as the solvent in early extractions and Skellysolve B was used later on. It was found as experimentation progressed that Skellysolve B possessed considerable advantage over trichloroethylene despite the fact that the percentage extraction seemed to be lower in the case of Skellysolve B (see Appendix).

The crude obtained through the use of trichloroethylene always bore a dark brown or chocolate color. There could be two reasons for this: (1) trichloroethylene has the ability of extracting colored bodies along with the desired extractables, and (2) considerable pyrolysis of trichloroethylene and wax occurs near the boiling point of trichloroethylene which is the temperature of distillation of the solvent. Experiments were carried out in an effort to see whether a white crude could be obtained by removing the solvent through the use of vacuum distillation. Although the method showed promise, no desirable results were obtained. Steam distillation was not tried.

The percent crude extracted from the bran as received is plotted against time in Figure 2 using trichloroethylene as the solvent for temperatures of 150° F. and 170° F. These curves agree remarkably well with those presented by Hub which leads the author to believe that agitation does not increase the efficiency of extraction. Hub's curves are plotted for no agitation.

Figure 3 presents a breakdown of the 150° F. curve of

Figure 2 into its two component curves, those of wax and oil. The wax and oil curves shown on this figure are subject to larger than normal experimental error and consequently they could be reversed from that shown or they could resolve into one common curve. The latter alternative seems to be the more likely one since the percentage of wax and oil extracted from the bran as received at moderate temperatures is in the ratio of one to one. Figure 4 presents the breakdown of the 170° F. curve and shows that the total extractables (the crude) at this high temperature contains more oil than wax. A temperature of 170° F. has been claimed to be the maximum safe temperature for the extraction of wax since the wax shows signs of decomposing above this temperature (8).

Removing all traces of trichloroethylene from the crude presented a difficult problem. Its molecular weight is relatively high when compared with that of Skellysolve B (hexane) causing its vapor pressure to be lower than that of Skellysolve B. Its high boiling point, 87.2° C. (185° F.), causes the pyrolysis of much of the crude. All of these factors led the author to abandon the use of trichloroethylene as the solvent in favor of Skellysolve B. Skellysolve B, a commercial grade of hexane, finds wide use as the solvent employed in the extraction of soybean oil and cotton-seed oil (3, 4).

Figures 5, 6, 7, and 8 depict in graphical form the data

collected for the extraction of the bran as received using
Skellysolve B as the solvent. Again, as in the extraction
results using trichloroethylene, the effect of an increase
in temperature is to increase the amount of crude extracted.
A maximum temperature of from 140-145° F. approaches too
close to the normal boiling point. A comparison of Figures
4 and 5 shows that as far as maximum percentage extraction
is concerned, trichloroethylene is better than Skellysolve B.
This advantage is offset considerably by the fact that it is
much easier to work with Skellysolve B than with trichloro-
ethylene. Skellysolve B also yields a much superior pro-
duct to that produced using trichloroethylene.

An interesting relationship existing between the oil-to-
wax ratio and temperature is brought out by a study of Figures
6, 7, and 8. At room temperature, 83° F., the ratio of oil to
wax is very high; at 103° F., the ratio has fallen consider-
ably; and at temperatures between 130 and 140° F., the ratio
is less than one indicating that now wax is the predominant
component of the crude.

Thus, in general, it may be postulated that the ratio of
oil to wax decreases as the temperature increases, or that
the ratio of wax to oil increases as the temperature in-
creases. The selectivity of wax over oil is then dependent
upon temperature. Whether the solvent used has any effect on
this selectivity cannot be said. Hub does not present break-

down data of his curves for trichloroethylene.

Fraction I, the pure bran fraction resulting from the additional separation of the bran as received, gave excellent results. From an inspection of the curves of Figures 10 and 11 it is seen that the bulk of the crude extracted from fraction I is wax. This is exactly the results sought for. In Figure 9 where the temperature is very low, the oil to wax ratio is high just as in Figure 6. Such low temperatures are of no value in commercial extraction units and are included solely as a means for comparing the oil to wax ratios at both low and high temperatures. At the higher temperatures of Figures 10 and 11, the oil to wax ratio is low; it is even lower than that for the bran as received at high temperatures. An appreciable amount of oil is still present in the extractables.

The effects of a variation in solvent ratio are shown graphically in Figure 11. In general, although an increasing solvent ratio increases the percentage extraction, an economic balance must be made between the selling price of the added product and the corresponding cost of the added solvent. The amount of oil extracted seems to remain constant with changes in the solvent ratio as shown in the figure.

The bulk of the oil contained in the bran as received is transferred to fraction II upon separation as shown by Figures 12 and 13. This seems logical when one reflects

that fraction II is made up of the germ and endosperm fractions of the whole grain which constitute the main oil bearers of the grain. Thus, even at high temperatures, the oil to wax ratio is very low and is in direct contrast to the behavior of fraction I and the bran as received. The results of the extractions made upon fraction II are of no direct value to the project at hand, but merely serve to show that if the grain used had been milled properly in the first place, a higher relative yield of wax to oil would have resulted because of the fact that the bulk of the oil would have been missing.

Figures 14 and 15 show the relative amounts of wax and oil extracted at 140° F. from the various fractions. From these figures it can be seen that fraction I yields the most wax and the least oil and is therefore the most ideal fraction of the three to extract. Hence, a prerequisite to attaining a predominance of wax over oil appears to be the proper milling of the grain into its various fractions.

ANALYSIS OF CRUDE SAMPLES

The procedure used in analyzing the extracted crude for wax and oil content is essentially that given in reference 1. Briefly, the procedure is as follows: 25 cc of acetone are added to the crude and the mixture is heated on a hot plate to promote solution of the crude. Twenty-five additional cc

of acetone are then added and the mixture is placed in a
refrigerator so that the temperature falls to at least 6° C.
The wax will crystallize out of the cold solution leaving
the oil still in solution. After an appreciable time inter-
val, the mixture is vacuum filtered through a previously
weighed asbestos-lined Gooch crucible so as to separate the
wax from the remaining miscella. The filtering apparatus
is pictured on the right-hand side of Plate III. The oil-
solvent solution is then evaporated to near dryness. After
allowing considerable time to insure thorough drying of the
wax and oil samples, the weights are recorded on the data
sheet presented in the Appendix.

Retention Ratio Equipment

A. Water bath

B. Extraction tube

C. Thermo-regulator No. 30142

D. Thermometer for bath (0-230° F.)

E. Knife heater for bath (250 watt, 115 V.)

F. Agitator No ST2

G. Miscella receiver graduate

H. Solvent entry graduate and thermometer

J. Kodak timer

K. Transformer (220-125 V.)

L. Mercury contact relay (115 V. contact; 25 amp;
     24 V. coil)

M. Transformer (125-24 V.)

N. Rheostat

P. Solvent reservoir

PLATE I

C 03200
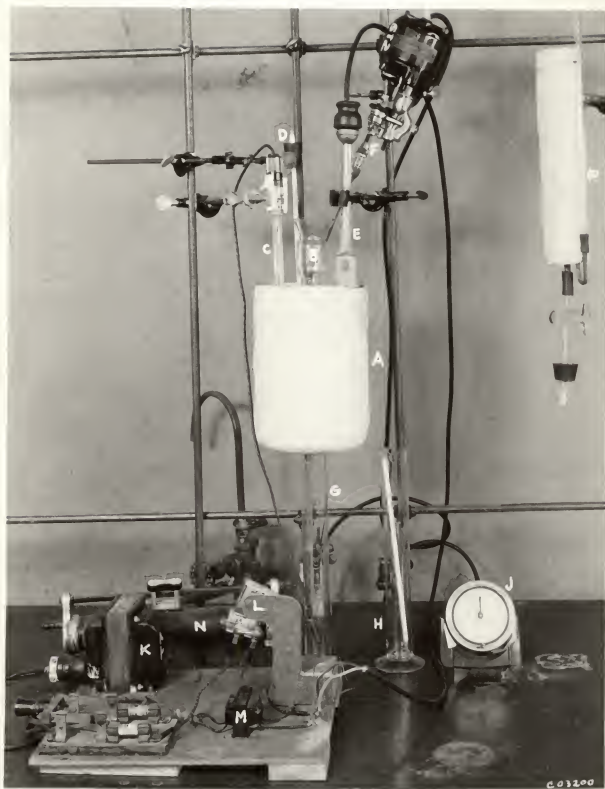
Extraction Equipment

A. Extraction vessel

B. Agitator No. ST-1

C. Knife heater (250 watt, 115 V.)

D. Thermometer (0-230° F.)

E. Variac (0-260 V.)

PLATE II

C03/99
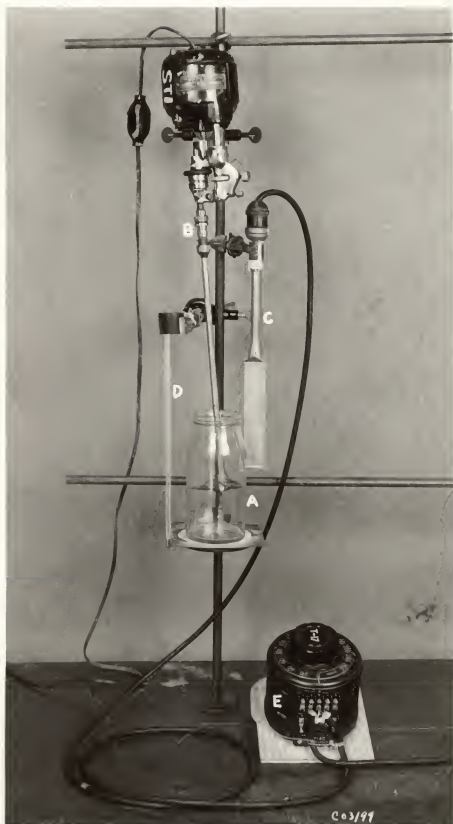
EXPLANATION OF PLATE III

Soxhlet Extractor and Crude Separation Equipment

A. Vacuum desiccator

B. Asbestos-lined Gooch crucible

C. Evaporating dish

D. Two-way stopcock

E. Safety flask

F. Soxhlet extractor

G. Soxhlet thimble

H. Hot plate

PLATE III
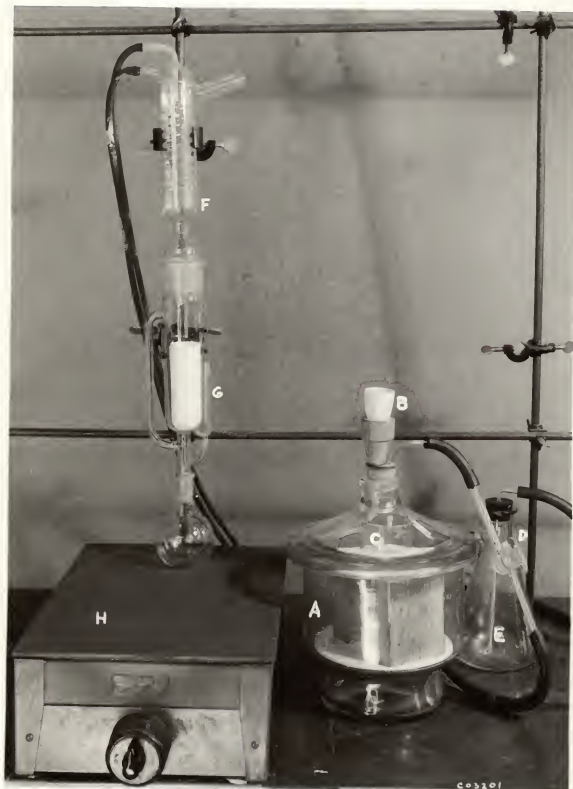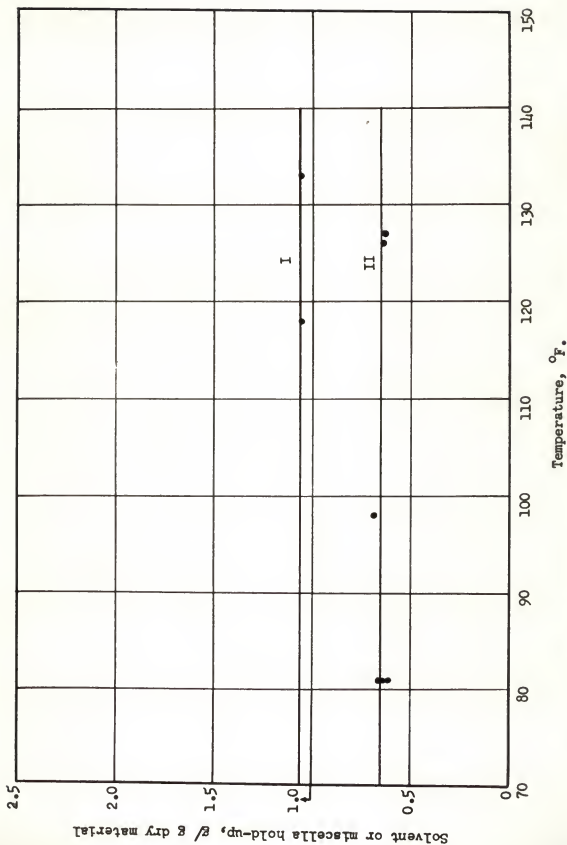
Fig. 1. Solvent or miscella hold-up in g/ g dry material versus temperature in °F.
Curve I is for trichloroethylene as the solvent; curve II is for Skelly-
solve B as the solvent. The material used is the bran as received.
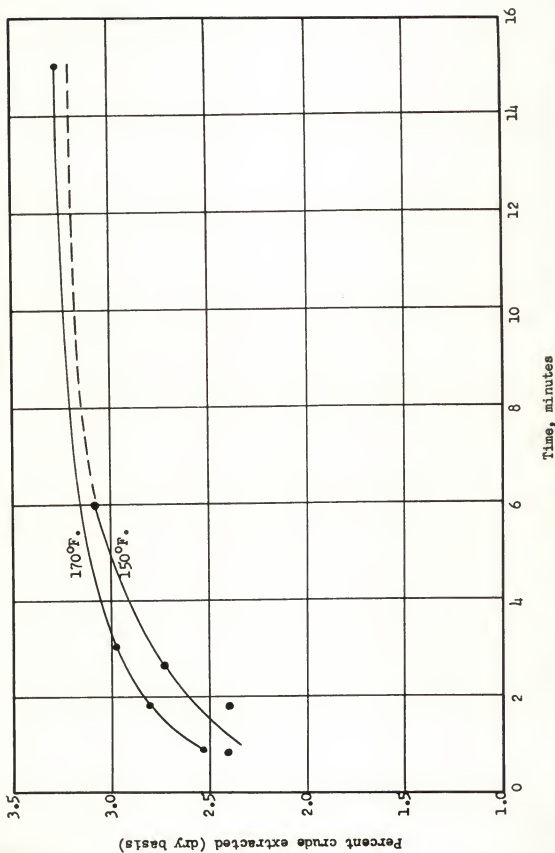
Fig. 2. Percent crude, dry basis, extracted from the bran as received versus extraction time in minutes using trichloroethylene as the solvent at 170°F. and 150°F. and a solvent ratio of 21.1 g solvent/g dry material.
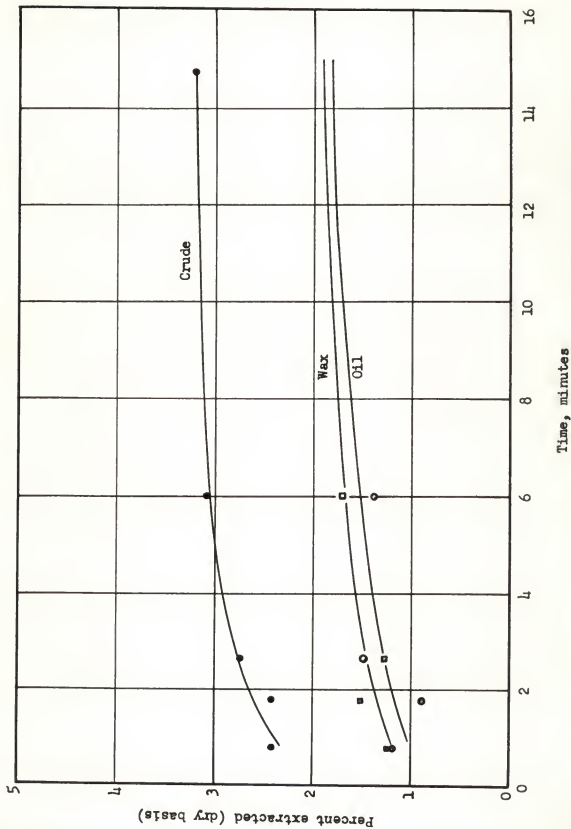
Fig. 3. Percent crude, oil, and wax extracted from the bran as received (dry basis) versus extraction time in minutes using trichloroethylene as the solvent at 150°F. and a solvent ratio of 21.1 g solvent/ g dry material.
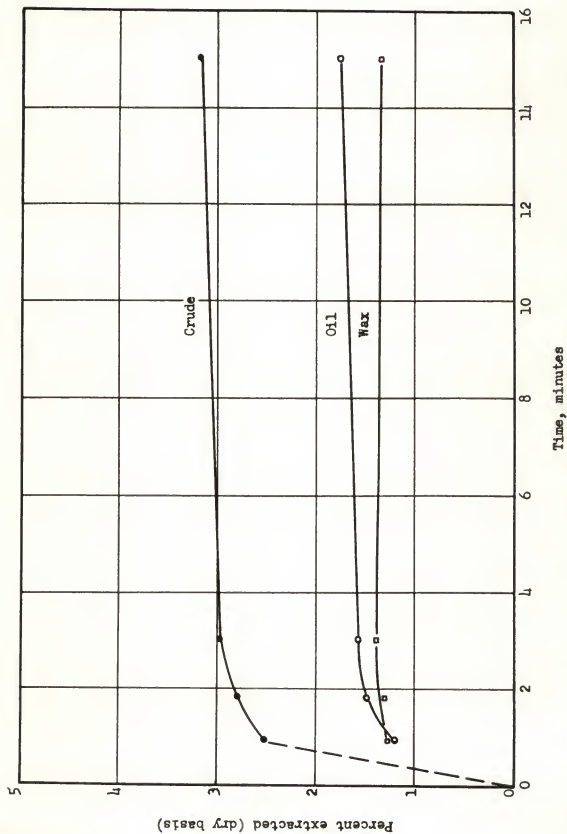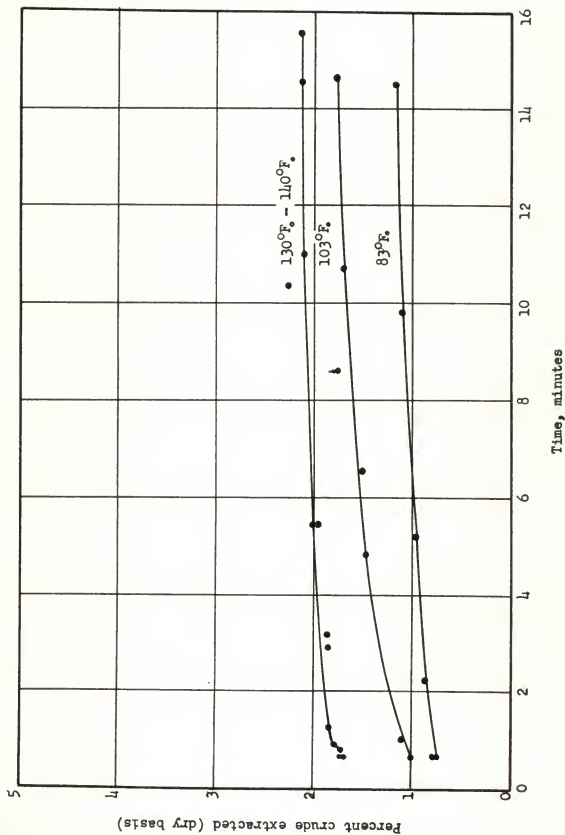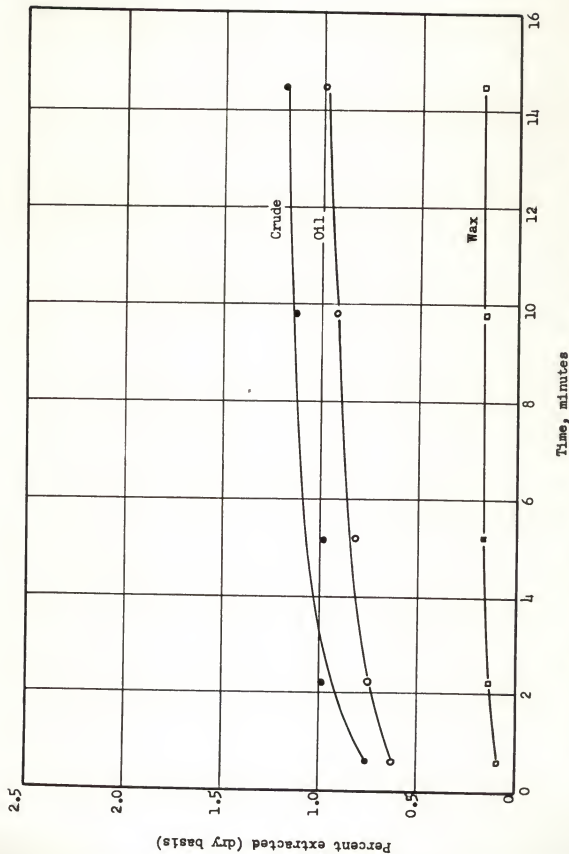
Fig. 4. Percent crude, oil, and wax extracted from the bran as received (dry basis) versus extraction time in minutes using trichloroethylene as the solvent at 170°F. and solvent ratio of 21.1 g solvent/ g dry material.

Fig. 5. Percent crude extracted from the bran as received (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at various temperatures and solvent ratio of 10.47g solvent/ g dry material.

Fig. 6. Percent crude, oil, and wax extracted from the bran as received (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at 83°F. and a solvent ratio of 10.47 g solvent/ g dry material.
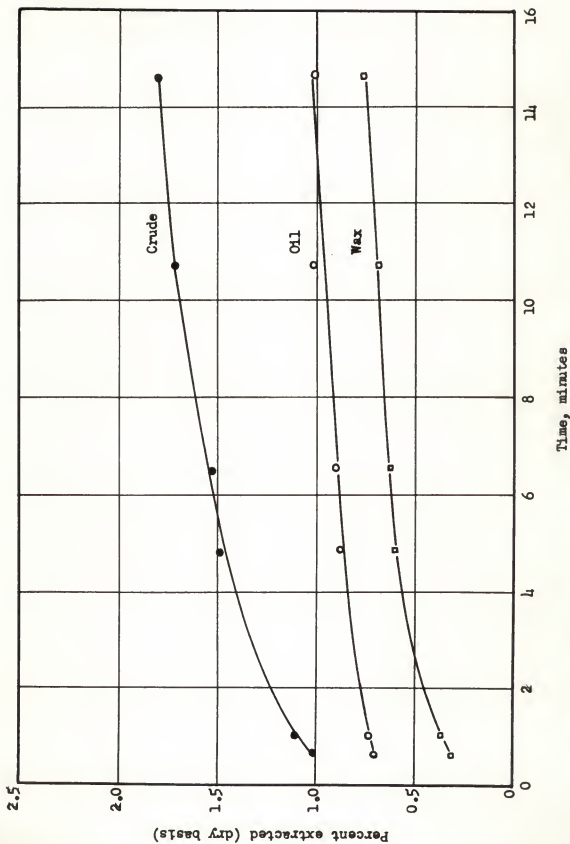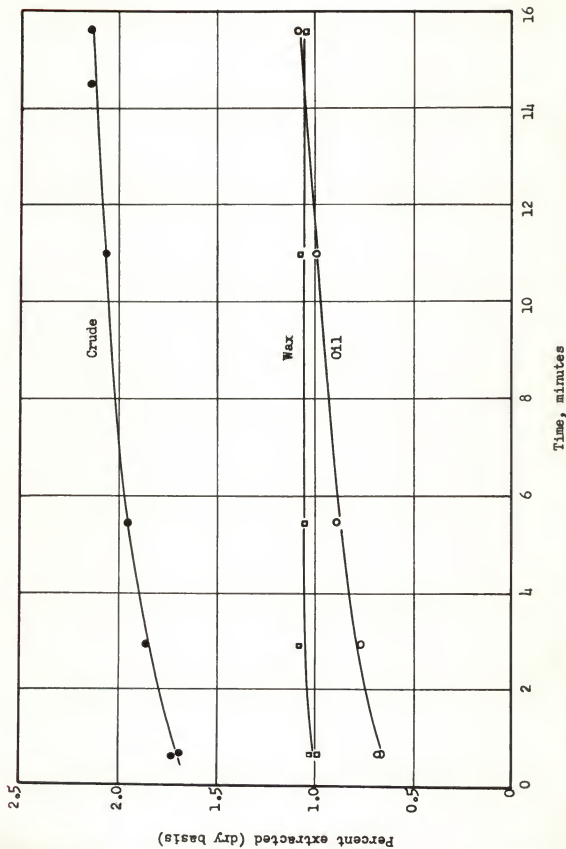
Fig. 7. Percent crude, oil, and wax extracted from the bran as received (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at 103°F. and a solvent ratio of 10.47 g solvent/ g dry material.

Fig. 8. Percent crude, oil, and wax extracted from the bran as received (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at temperatures of 130°F. and 140°F. and solvent ratio of 10.47.
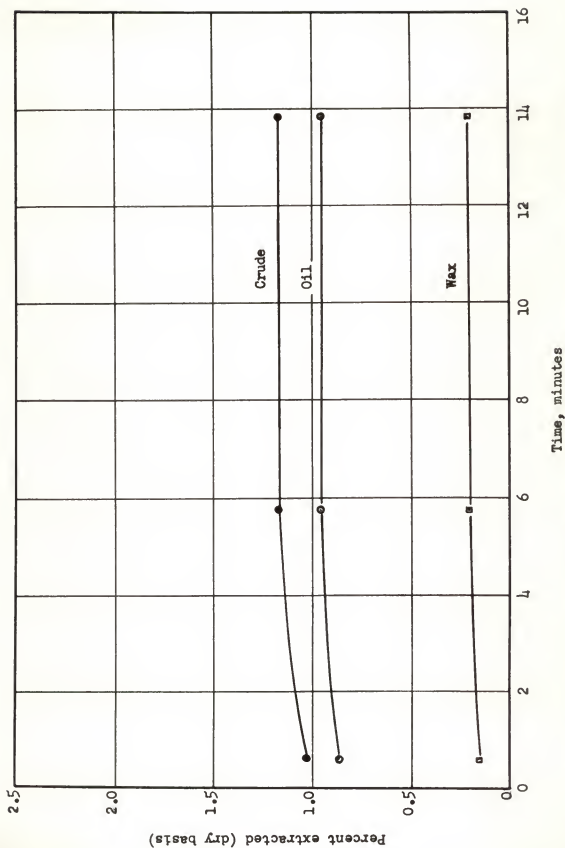
Fig. 9. Percent crude, oil, and wax extracted from fraction I (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at 80°F. and solvent ratio of 10.47 g solvent/g dry material.
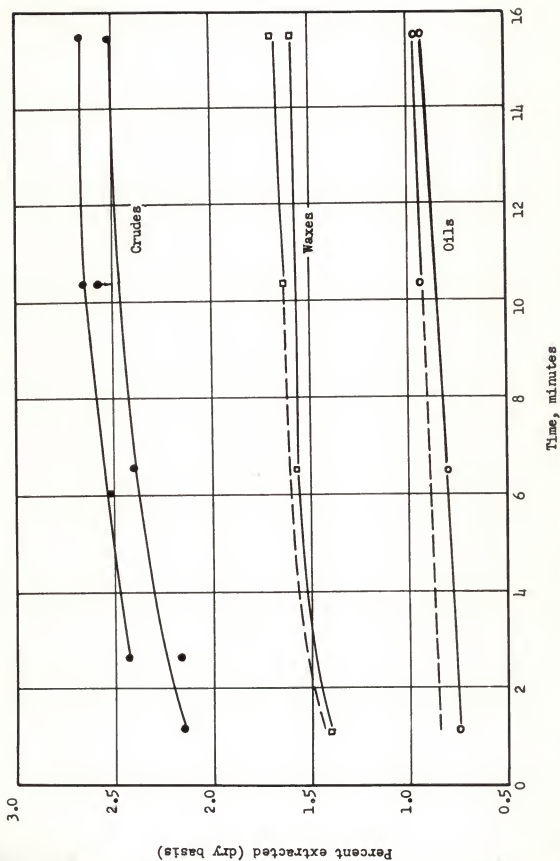
Fig. 10. Percent crude, oil, and wax extracted from fraction I (dry basis) versus ex-
traction time in minutes using Skellysolve B as the solvent at a solvent ratio
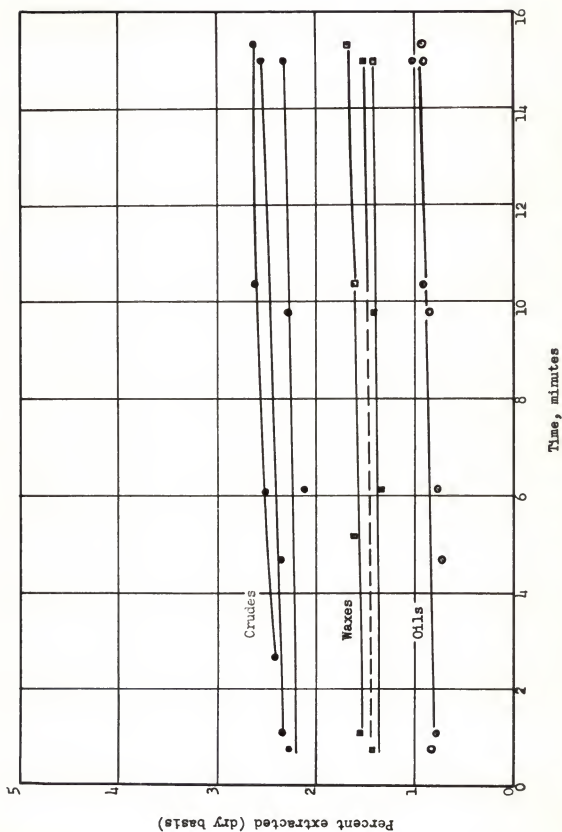of 10.47 g solvent/ g dry material. Upper curves for 140°F.; lower, 120°F.

Fig. 11. Percent crude, oil, and wax extracted from fraction I (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at 140°F. Solvent ratios: upper curves each group, 10.47; center, 7.91; bottom, 5.24.
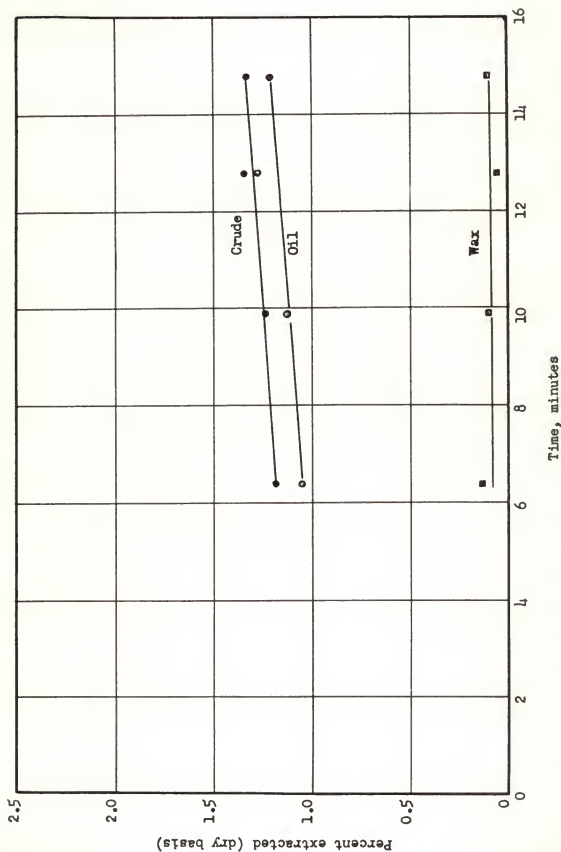
Fig. 12. Percent crude, oil, and wax extracted from fraction II (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at 80°F, and a solvent ratio of 10.47 g solvent/ g dry material.
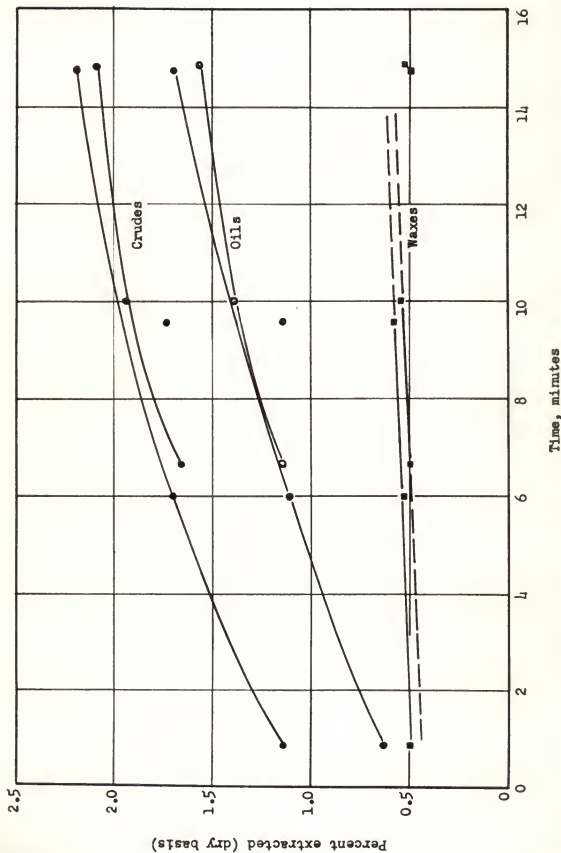
Fig. 13. Percent crude, oil, and wax extracted from fraction II (dry basis) versus ex-
traction time in minutes using Skellysolve B as the solvent at a solvent ratio
of 10.47 g solvent/ g dry material. Upper curves for 140°F.; lower for 120°F.
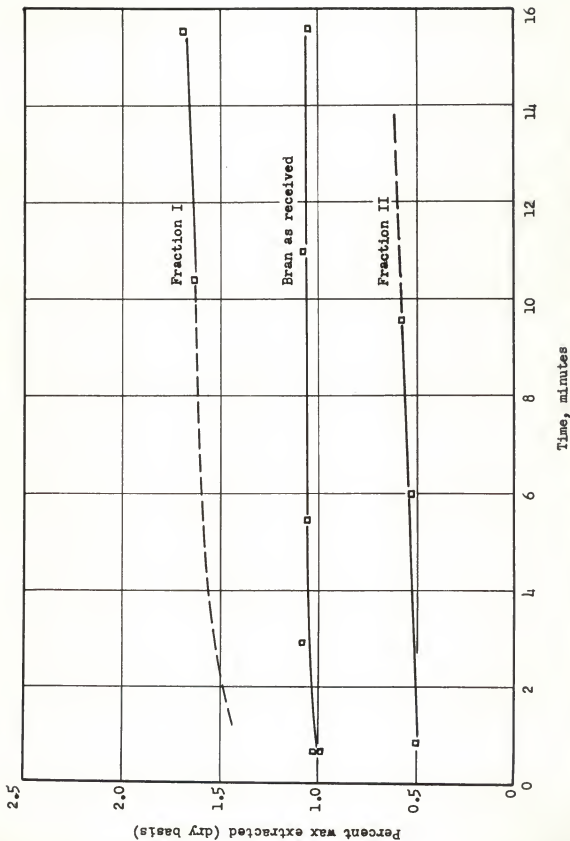
Fig. 14. Comparison of the percent wax extracted from the bran as received, fraction I, and fraction II (dry basis) versus extraction time in minutes using Skelly-solve B as the solvent at 110°F, and a solvent ratio of 10.47.
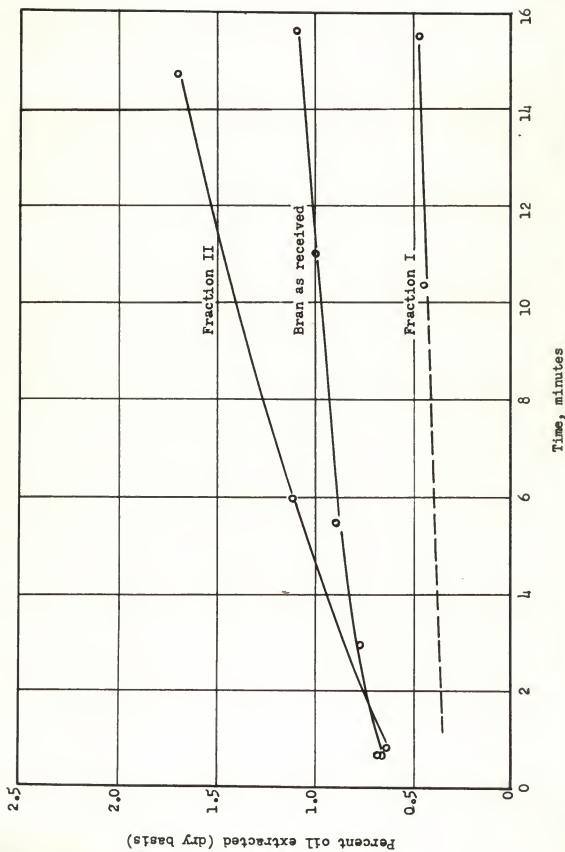
Fig. 15. Comparison of the percent extraction of oil from the bran as received, fraction I, and fraction II (dry basis) versus extraction time in minutes using Skellysolve B as the solvent at 140°F. and a solvent ratio of 10.47.

THE KENNEDY EXTRACTOR

The author has examined the literature for possible
types of extraction apparatus for use in the wax extrac-
tion of sorghum bran. Among the different types of ex-
traction apparatus considered, one type seemed to be out-
standing for this particular project. This piece of equip-
ment is named for its inventor, Mr. Angus B. Kennedy.

The Kennedy extractor (3, 4, 7, 10) is probably the
most versatile extractor for the extraction of vegetable
oils. It is manufactured by the Vulcan Copper and Supply
Co., Cincinnati, Ohio. Two Kennedy pilot plants are known
to the author. One is located at the company already men-
tioned and the other is located at the Northern Regional
Research Laboratory in Peoria, Illinois. A schematic dia-
gram of the unit is presented in Figure 16. Reference 4
contains an excellent photograph of a one hundred ton unit
installed at Abilene, Texas.

The following excerpt (4) is given in connection with
Figure 16:

> The continuous extractor consists of a series
> of enclosed extraction chambers or sections: into
> each of these is fitted an impeller wheel assembly
> for moving the solid materials through the liquid.
> Each impeller wheel assembly consists of an enclosed
> hub (the lower portion of the hub is partially sub-
> merged in the liquid), four curved blades of per-
> forated metal, and a shaft extending through bear-
> ings in the sides of the extractor walls. The im-
> peller wheel assemblies are driven from the outside
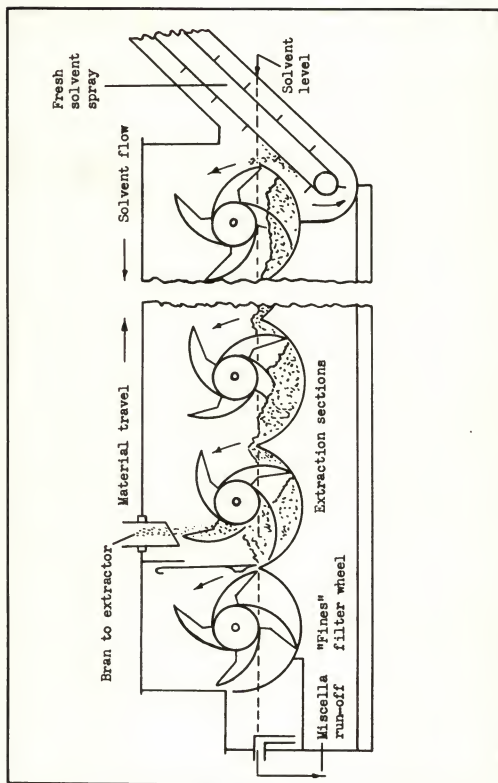> by a common drive shaft through a worm and gear
> arrangement.

Fig. 16. Cross-sectional view of the Kennedy extractor.

The movement of the solid material through the extractor is accomplished by means of the impeller wheels. Note the direction of rotation of the impellers as indicated in the diagram. As the solids are carried up through the liquid portion of each extractor section, they are compressed slightly between the curved wall and the impeller blade. This slight compression serves to reduce the amount of entrained liquid carried over into the next section. When the blade has carried the solids up through the liquid, the solids are sloughed off into the next section.

A definite cycle is followed by the solid material in passing through the Kennedy extractor. The first step of the cycle is dispersion. This step occurs when the solid wedge which sloughs off the impeller blade strikes the surface of the liquid in the next section. Dispersion is followed closely by immersion as the solid is immersed throughout the liquid phase. Movement through the liquid, compression, and draining constitute the final steps and form the basis for the intimate contact which exists between the solvent and solid particles.

The versatility of the Kennedy extractor has led the author to its selection as a possible unit for the successful extraction of wax from sorghum bran. In tests carried out on the two pilot plants cited, it was found that the extractor worked equally well with granular, powdery, gelatinous, fibrous, stringy, and pulpy material as it did with

the conventional flake form of material. In addition, a
large variety of solvents have been successfully used
among them being commercial hexane and trichloroethylene.

DESIGN OF THE EXTRACTOR UNIT

The familiar graphical solution to extraction problems
was used to determine the number of ideal stages that would
be required to effect a given separation. The procedure
and nomenclature used are covered adequately in reference 2.
For convenience, the nomenclature used on the diagram is re-
peated here.

- x Fractional composition of the bottom stream in
     terms of the component designated by a subscript.
     In this case A refers to solute or the total
     crude (wax and oil), and s to the solvent,
     Skellysolve B.
- y Fractional composition of the extract stream in
     terms of the component designated by a subscript.

The numerical subscripts refer to the stage number
where n is the last stage. The n 1 th stage is an imagi-
nary stage from which the solvent is introduced to stage n;
similarly the 0 th stage is an imaginary stage from which
the solids are introduced to stage 1. The line EG" is de-
termined by the retention ratio, and since the ratio of
solvent retained to solid is to be considered constant,
this line when extended would meet the two extended sides
of the right triangle at the missing corner. If the line
EG" were drawn parallel to the hypotenuse of the triangle

(top line), the ratio of miscella retained to solid would remain constant. The assumption that these two ratios are equal has been discussed in detail in the section covering the determination of the retention ratio.

Figure 17 presents the graphical solution using Skelly-solve B for two different solvent ratios, 7.91 and 1.2. The data collected for runs lasting fifteen minutes were used in determining the amount of extraction which could be expected. This expected extraction came out to be about 90 percent. Primed quantities on the diagram refer to a solvent ratio of 7.91; unprimed quantities to a solvent ratio of 1.2. The temperature of extraction is 140° F.

The results of the figure show that at the high solvent ratio only one stage would be necessary to give the desired extraction, but that at the lower solvent ratio, a total of four theoretical stages are necessary. Thus, a Kennedy extractor having four ideal stages could extract 90 percent of the extractables from fraction I at a solvent ratio of 1.2, a temperature of 140° F., and a contact time of somewhat over fifteen minutes.

ECONOMICS OF WAX EXTRACTION

It has been stated that the aim of this project is toward the successful operation of some selected low capacity extraction unit for extracting the wax from sorghum bran.
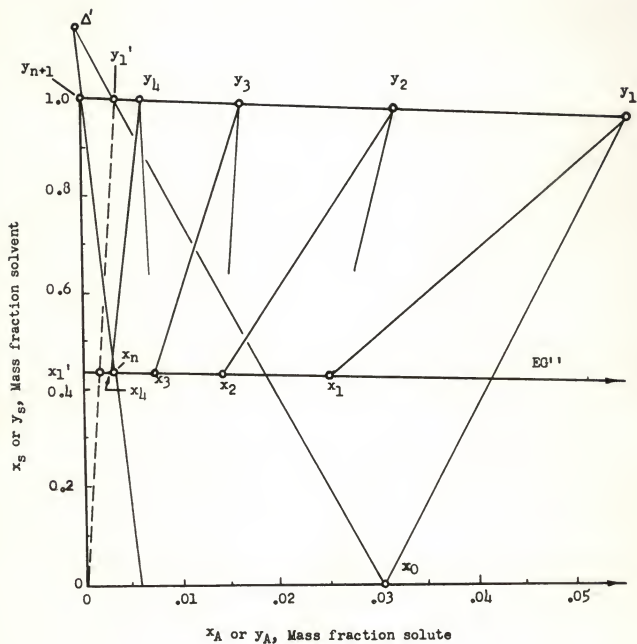
Fig. 17. Graphical solutions of counter-current multiple contact extraction of wax and oil (solute) from fraction I for solvent ratios of 7.91 and 1.2.

This statement brings up the question, "What is meant by
the term low capacity?" It is difficult to predict with
any great accuracy the ideal capacity of an extraction
unit. The capacity of the Kennedy unit can be varied at
will be increasing the input rate and inserting extraction
sections to compensate for the loss in efficiency. However,
it is a known fact that, in general, the unit cost operation
increases as the capacity decreases. It is considered
economically undesirable to build extraction apparatus
having capacities of less than 50-75 tons per day (11).

The extraction process can be carried out in one of
two ways: (1) each mill in which the whole grain is separ-
ated into its respective fractions could have a Kennedy ex-
traction unit, or (2) the aggregate bran fraction from
several mills in a certain area could be sent to one common
Kennedy extraction unit conveniently located with respect to
each mill. From discussions with Dr. H. N. Barham, Professor
in the Chemistry Department, a good figure for the capacity
of any individual mill per day would be about three carloads[2]
of whole grain. An average analysis of several types of
sorghum grain is as follows:

> Bran..........8 percent
> Endosperm.....76 percent
> Mill fines....4 percent
> Germ..........12 percent

---

2   One carload of sorghum grain weighs approximately
40,000 pounds.

Using these figures, the capacity of a Kennedy unit located at an individual mill would be only 4.8 tons per day. Of course, such a low capacity is out of the question for economical reasons. Thus, it is evident, that in order to operate in an economical manner, one common Kennedy extractor should be provided for a group of mills. If, however, all whole sorghum grain is shipped to one common mill, the ideal location of the Kennedy unit would be at that mill. Such a common mill might process at least ten carloads of whole grain per day corresponding to a capacity of sixteen tons for the Kennedy unit. In order to include a factor providing for more capacity, a good estimate for a low capacity Kennedy unit would seem to be around twenty-five tons per day. Although even this capacity is well below the minimum general economical capacity stated above (11), it is hoped that the unit can be operated at a profit.

The actual capacity decided upon in the long run will have to be directly dependent upon the availability of material (bran). Since the economics of the overall sorghum picture is not dependent upon the wax extraction step, but rather upon the demand for the endosperm of the grain, variations in price and demand of the endosperm, from which starch is extracted, will have a direct influence on choosing the correct capacity for a Kennedy extractor.

The cost analysis which follows is not intended to be an accurate analysis. The figures given are, for all practical

purposes, merely estimates and are based for the most part
upon those given in reference 4 which are for a 120 ton
plant and apply to the year 1941. In this analysis the
cost of sorghum bran is conveniently taken as the cost of
whole grain sorghums. The solvent used is Skellysolve B.

Table 3. Processing cost analysis for a 25 ton plant[3] (1951).

	Preparation and extraction		
	Item of expense	Investment	Yearly charges
1.	Land, int. at 5%	$ 935[4]	$ 47
2.	Building, int. at 5%	10,700[5]	535
3.	Building, depr. at 5%		535
4.	Building, maint. at 2%		214
5.	Office and furn., int. at 5%	1,250[5]	63
6.	Office and furn., depr. at 5%		63
7.	Office and furn., maint. at 4%		50
8.	Machinery, piping, wiring, etc., installed, int. at 5%	60,000[5]	3,000
9.	Depr., 10% of item 8		6,000
10.	Maint., 16¢ per. bu.		4,000
11.	Solvent inventory	3,500[6]	175
12.	Taxes, $30 per M on 70% valuation, on items 1,2,5,8,11		1,605
13.	Ins., $20 per M on 90% valuation on items 2,5,8,11		1,358
14.	Steam, 35¢ per M lb.[7]		2,880
15.	Water, 5¢ per 100 cu. ft.		800
16.	Power, 1.33¢ per Kw-hr.		2,000
17.	Solvent loss, 14.5¢/gal.		2,000
18.	Labor, 2 men per shift, at $1.20 per hr.		17,300
19.	Superintendent		7,000

Table 3. (concl.)

	Item of expense	Investment	Yearly charges
20.	Oil storage tanks, installed, 10,000 gal., int. at 5%	$ 1,750	$ 88
21.	Depr., 5% of item 20		438
22.	Maint., 2% of item 20		175
23.	Oil inventory, 1 wk. @ 20¢ per lb.	21,000	1,050
24.	Wax inventory, 1 wk. @ $1.30 per lb.	91,000	4,550
25.	Taxes, $30 per M on 70% valuation on items 20,23,24		2,535
26.	Ins., $20 per M on 90% valuation on items 20,23,24		2,174
	Totals	$190,135	$60,635

Credits on a yearly basis [8]

27.	Oil sales [9]		$ 90,000
28.	Wax sales [10]		390,000
	Total		$480,000

29. Profit: $55 per ton of bran extracted.

---

3 Reference 4, pp. 86-87. All figures multiplied by 0.208 to reduce from 120 ton to 25 ton capacity.
4 Factor of 1.5 adapts 1941 data to 1951.
5 Factor of 2 adapts 1941 data to 1951.
6 Based on 24,000 gal. inventory at 14.5¢ per gal., Oil, Paint, and Drug Reporter, June 25, 1951.
7 Price of utilities is considered unchanged.
8 A total of 7500 tons of bran per working year is assumed to be extracted. The cost of the bran is assumed to balance the selling price of the meal.
9 Based on 20¢ per lb. given for peanut oil in Oil, Paint, and Drug Reporter, June 25, 1951.
10 Based on $1.30 per lb. given for carnauba wax in Oil, Paint, and Drug Reporter, June 25, 1951.

It must be borne in mind that the above analysis is very crude. However, the figures seem to indicate that the solvent extraction of wax from sorghum bran can be an economical process. The cost of milling the whole grain and separating the wax from the oil is not included in the above analysis.

RECOMMENDATIONS

1. The recommendation is made that all small laboratory work be ceased except possibly the running of a few experiments based on determining whether a better separation of wax and oil could be obtained by subjecting the bran to a cold extraction followed by a hot extraction. It was brought out in the text of this paper that the oil to wax ratio definitely decreased with rising temperature and that oil was by far the predominant component of the crude at low temperature. This fact suggests the possibility of a cold extraction to remove the bulk of the oil followed by a hot extraction to remove the wax.

2. A Kennedy unit should be built in the main laboratory on a pilot plant scale. In its operation properly milled bran should be used as the solid material and Skellysolve B should be used as the solvent.

CONCLUSIONS

1. The retention ratio or solvent hold-up of sorghum bran is a constant and hence does not vary with temperature or time of contact.

2. The oil-to-wax ratio resulting from the extraction of sorghum bran from poorly milled sorghum grain is bound to be undesirably high.

3. The oil-to-wax ratio of the crude resulting from the extraction of sorghum bran decreases with an increase in the temperature.

4. The amount of crude extracted is a function of the temperature, contact time, and the solvent ratio and increases as each of these variables is increased.

ACKNOWLEDGMENTS

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appreciation for the invaluable aid
given him by Dr. G. David Shilling,
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project. Appreciation is also ex-
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and to Dr. H. N. Barham, Professor of
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APPENDIX

Trichloroethylene vs. Skellysolve B

The relative merits of trichloroethylene and Skelly-
solve B are given as follows based upon the actual experi-
ence of the author with both of these solvents.

Trichloroethylene

1. Non-flammable
2. Fumes are very toxic and have a pungent odor
3. Extremely difficult to remove from crudes extracted
4. Resulting crude and wax a deep chocolate color
5. High boiling point causes pyrolysis
6. Expensive
7. Gives a high percentage yield
8. Wax remains dissolved in cold solution

Skellysolve B

1. Highly flammable
2. Fumes are mildly toxic and have a pleasant odor
3. Easily removed from crudes extracted
4. Resulting crude yellow-ish-white in color; white wax
5. Low boiling point depresses pyrolysis
6. Inexpensive
7. Gives a lower percentage yield than trichloro-ethylene (reason: the lower extraction tempera-ture which must be used)
8. Most of wax crystallizes from cold solution

Calculations

A sample data sheet designed for the determination
of the retention ratio is presented on the following page.
The volume of solvent used for each run was 130cc. The
volume of miscella obtained was recorded at intervals of
one minute until dripping ceased for all practical pur-
poses. The temperature of the miscella was recorded and
the volume corrected to the initial extraction temperature
volume with the use of a specific gravity versus tempera-
ture chart for the particular solvent (Figures 18 and 19).

This volume plus about three or four cc to account for lossage was subtracted from the initial solvent volume to give the volume of solvent retained. The solvent hold-up was then directly calculated as shown on the data sheet.

A sample data sheet used for the analysis of the extraction runs is also presented in this section. This data sheet is self explanatory.

DATA SHEET

Date_____, 1951_____   Run_____

Wt. of bran used_____gm_   Moisture content_____

Temperature_____   Solvent vol. used_____cc

                    Solvent_____

Volume of miscella obtained: Time

At closing of solvent clock _____cc   _____

1 minute later   _____cc   _____

2 minutes later   _____cc   _____

3 minutes later   _____cc   _____

4 minutes later   _____cc   _____

5 minutes later   _____cc   _____

Time dripping ceases for

   all practical purposes   _____cc   _____

RUN ANALYSIS:

        Volume of solvent used   _____cc

        Volume of 'solvent' recovered   _____cc

            Volume retained   _____cc

            Weight retained _____gm; sp. gr._____

        Solvent Hold-up_____gm/gm of bran (dry basis)

If analysis of miscella is desired attach appropriate
data sheet.

RUN ANALYSIS

Run No._____   Date of Run_____

Temperature_____   Time_____

Solvent_____   Solvent Volume_____

  Wt. of Bran Used_____   Moisture Content_____

Wt. of Oil + Wax + Flask_____gm   Wt. of Wax + Flask_____gm

               - Flask_____gm   - Flask_____gm

Wt. of Oil + Wax   _____gm   Wt. of Wax in Flask_____gm

Wt. of Wax + Crucible_____gm   Total Wt. of Wax_____gm

           - Crucible_____gm   Wt. of Oil + Dish_____gm

Wt. of Wax in Crucible_____gm   - Dish_____gm

                         Wt. of Oil   _____gm

     Wt. Balance: Oil + Wax   _____gm

                    Oil _____gm

                    Wax _____gm

                   Total   _____gm

       Wt. of Dry Bran_____ - _____

              = _____gm

        % Oil + Wax _____%

        % Oil   _____%

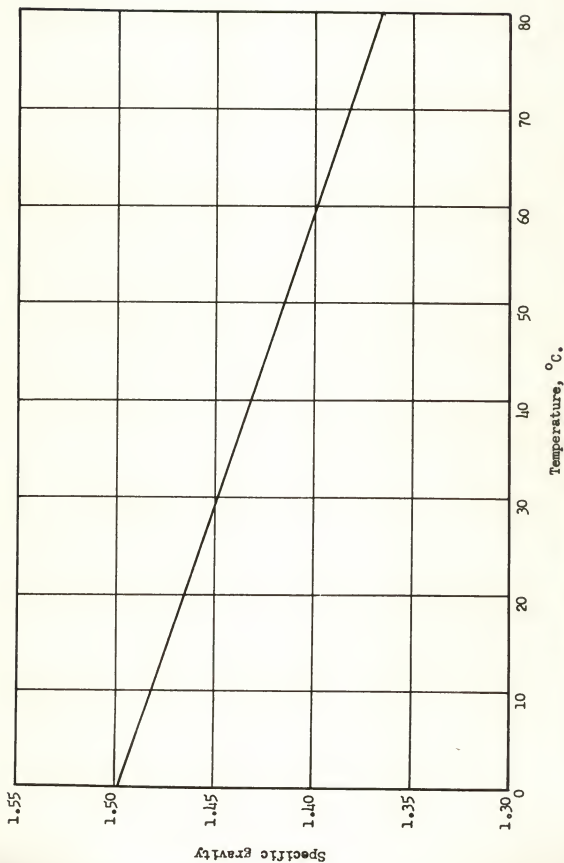   % Wax   _____%

Fig. 18. The specific gravity of trichloroethylene (boiling point 87.2°C.) versus temperature in °C.
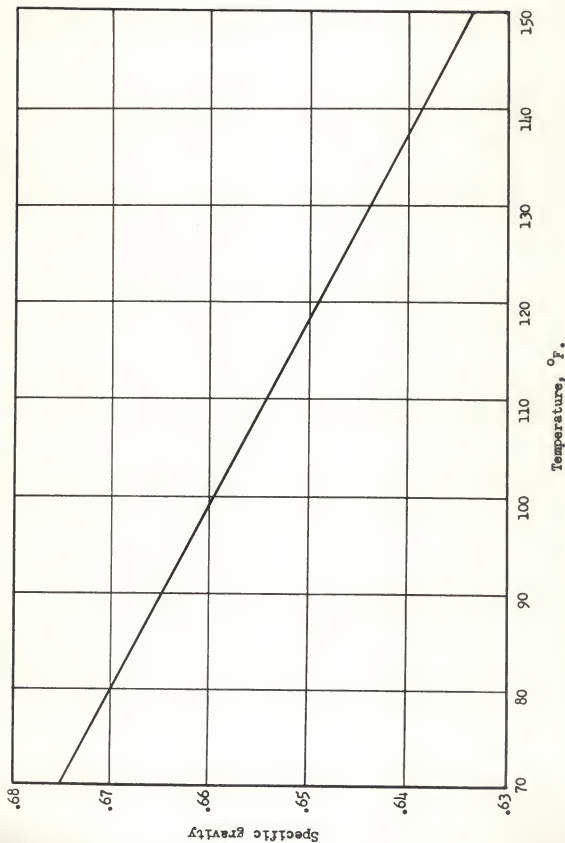
63

Fig. 19. The specific gravity of Skellysolve B (boiling point 152.6°F.) versus temperature in °F.

THE SOLVENT EXTRACTION OF WAX
FROM SORGHUM BRAN

by

ROBERT JAMES KEHM

B. S., Washington University, St. Louis, 1950

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AN ABSTRACT OF A THESIS

submitted in partial fulfillment of
the requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE COLLEGE
OF AGRICULTURE AND APPLIED SCIENCE

1951

The ultimate aim of this project is the selection and successful operation of a low capacity extraction unit suitable for extracting the wax from sorghum bran. In working toward this goal, the investigations which were begun by K. A. Hub were considerably extended and modified. This work included the effect of temperature, contact time, and solvent ratio upon the percentage crude extracted. The author has carried out this investigation using Skellysolve B as a solvent whereas previous work was confined to using trichloroethylene as the solvent. The author has also presented breakdown curves of the crude (total extractables) curve into its two component curves, that of wax and oil. These breakdown data have heretofore been lacking.

It was found, after considerable experimentation, that the bran previously used had not been properly milled and that the bulk of the weight could be attributed to grain fractions other than bran. The author made a more thorough separation of the bran and ran extraction experiments on each separated fraction. It was found that when the bran is properly milled, wax is the predominant constituent and oil is a secondary constituent. Excellent results were obtained with the pure bran fraction.

The Kennedy extractor was chosen as the most suitable type of extraction apparatus for the extraction of wax from sorghum bran because of its extreme versatility and its method of providing intimate contact between the solvent and the material being extracted. This extractor consists of a series of semi-circular extraction chambers each fitted with an impeller wheel. The solid material to be extracted is forced through the solvent in each section by the impeller and sloughed off into the next section. Solvent flows counter-current to the flow direction of the solids. From a graphical solution it was found that a Kennedy extractor having four theoretical stages could extract ninety per cent of the wax and oil from the pure bran fraction at 140° F., a solvent (Skellysolve B) ratio of 1.2, and a contact time of somewhat over fifteen minutes. In order to carry out this solution the retention ratio (weight of solvent retained per unit weight of bran) had to be determined. It was recommended that a Kennedy extractor be built in the main laboratory of the Chemical Engineering Building.

A crude economic analysis of a proposed twenty-five ton Kennedy unit indicated that the extraction process should be an economical one.

The conclusions derived from this work are as follows:

1. The retention ratio or solvent hold-up of sorghum bran is a constant and hence does not vary with temperature or time of contact.

2. The oil-to-wax ratio resulting from the extraction of sorghum bran from poorly milled sorghum bran is bound to be undesirably high.

3. The oil-to-wax ratio of the crude resulting from the extraction of sorghum bran decreases with an increase in the temperature.

4. The amount of crude extracted is a function of the temperature, contact time, and the solvent ratio and increases as each of these variables is increased.